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**CHEMICAL, PHYSICAL AND ENGINEERING
PERFORMANCE CHARACTERISTICS OF A NEW
FAMILY OF PERFLUORINATED FLUIDS**

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TECHNICAL REPORT AFML-TR-65-358

SEPTEMBER 1965

Each transmittal of this document outside the agencies of the U. S. Government must have prior approval of the Fluid and Lubricant Materials Branch (MANL), Nonmetallic Materials Division, Air Force Materials Laboratory, Wright-Patterson AFB, Ohio 45433.

AIR FORCE MATERIALS LABORATORY
RESEARCH AND TECHNOLOGY DIVISION
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

AFML-TR-65-358

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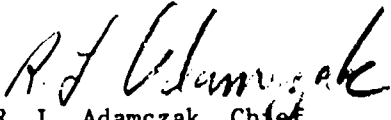
FOREWORD

This report was prepared by the Fluid and Lubricant Materials Branch, Nonmetallic Materials Division, Air Force Materials Laboratory, Research and Technology Division. Work was initiated under Project No. 7343, "Aerospace Lubricants", Task No. 734303, "Fluid Lubricant Materials", with Roland E. Dolle, Frank J. Harsacky, Herbert Schwenker and Robert L. Adamczak acting as project engineers.

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This technical report has been reviewed and is approved.


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ABSTRACT

A new class of high temperature fluids, designated by Du Pont code PR-143, has been extensively investigated to determine its potential in the area of advanced lubrication and energy transfer. The PR-143 fluids have exhibited excellent high temperature oxidative stability, a broad fluid range, good lubricity, and a high degree of fire-resistance; thereby making them promising candidates for aerospace systems of the future. In the design of future systems some shortcomings of these perfluorinated polymeric materials must be considered; namely, corrosion of certain metal alloys at high temperatures and their relatively high density.

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SECTION I

INTRODUCTION

With the advent of new high performance aerospace systems advanced fluids and lubricants are needed which are capable of withstanding stringent thermal and oxidative stress at temperatures exceeding 500°F without undergoing appreciable degradation. High thermal and oxidative stability must be accompanied by adequate low temperature fluid properties and satisfactory lubricating characteristics. Current available materials such as the super-refined mineral oils, diesters, and the polyphenylethers lack one or more of the important properties required. For example, the mineral oils possess good low temperature fluid properties, exceptional lubricating ability and are thermally stable at 700°F, but they do not have sufficient oxidative resistance at elevated temperatures. The diesters, while fulfilling current gas turbine engine oil requirements, cannot be expected to survive oxidatively above 400°F without modification resulting in appreciable sacrifice in low temperature properties. The polyphenylether neat fluids have demonstrated good oxidative stability at 500 to 550°F and are thermally stable at 700°F, but these materials have high pour points and are regarded as having relatively poor lubricating ability (References 1 and 2).

In the current search for advanced fluids, one of the targets has been to obtain a fluid that is comparable with polyphenylether in oxidative and thermal stability, but with a considerable improvement in low temperature fluidity and better lubricity.

In the fall of 1962 a polymeric perfluorinated high temperature fluid called PR-143 was received by the Nonmetallic Materials Division, Fluid and Lubricant Materials Branch (MANL) from E. I. Du Pont de Nemours and Company, Pioneering Applications Division, Organic Chemicals Department. Preliminary data obtained by Du Pont indicated that the fluid had high oxidative and thermal stability with improved low temperature fluid properties over polyphenylether. At that time a cooperative program was initiated by MANL and Du Pont to fully characterize this fluid in order to determine its potential for meeting advanced requirements of the U.S. Air Force in the area of lubricants and energy transfer fluids. Since the early investigations (Reference 3) PR-143 fluids have undergone extensive characterization, not only by MANL and Du Pont, but also by other governmental agencies and various contractors. The bulk of these studies, both laboratory experiments and actual performance tests are reported herein. Emphasis has been placed on the ability of the PR-143 fluids to function as hydraulic fluids, gas turbine engine oils and as base fluids for high temperature greases.

SECTION II

DISCUSSION

The physical and chemical properties, including thermal and oxidation stability, metal compatibility, and engineering performance characteristics of a series of PR-143 fluids were investigated. The difference in the types of PR-143 fluids examined was generally physical, rather than chemical, varying primarily in molecular weight distribution. The various PR-143 fluids and their common code designations are listed below:

PR-143M :	ELO-62-99
PR-143P :	ELO-63-70
PR-143, Lot I :	ELO-63-138, MLO-63-42, MLO-7705
PR-143, Lot II :	ELO-64-20, MLO-64-8
PR-143, Lot III :	ELO-64-21, MLO-64-9
PR-143, Lot V :	ELO-64-73, MLO-65-31
PR-143U :	MLO-63-63
PR-143W :	MLO-7723
PR-143X	
PR-143AB, Lot IV	
PR-143AC:	ELO-65-35, MLO-65-32

In the following discussion, the fluids will be referred to by the PR-143 designation assigned by Du Pont.

Many of the characteristics of the PR-143 fluids are compared with the polyphenylether, *m*-bis (*m*-phenoxyphenoxy) benzene (ELO-62-29) which will be referred to as simply 5P-4E. The 5P-4E is actually a mixture of isomers, but predominately meta.

GENERAL PHYSICAL AND CHEMICAL PROPERTIES OF PR-143 FLUIDS

Physical and chemical data for several PR-143 fluids are summarized in Table 1, together with similar data for 5P-4E for comparison. Additional characteristics of PR-143, such as radiation stability and elastomer compatibility, are found in Tables 2 through 4.

The results of various instrumental and chemical analyses of PR-143 fluids are given in Table 5.

Fluidity

The operating temperature range for many of the PR-143 fluids appears to be in the neighborhood of slightly above 0°F to + 700°F. This is based on the estimation

that for low temperature operation, a hydraulic fluid should have a viscosity no greater than 4000 centistokes. For gas turbine oils, the maximum allowable viscosity is about 13,000 centistokes. The high temperature viscosity for both areas of application should be no less than 0.5 centistokes. The improvement of the PR-143 fluids over 5P-4E in temperature-viscosity characteristics is demonstrated by comparing the viscosity index of the materials (115 V.I. versus -81 V.I.).

Viscosity-pressure data (Figure 1) for PR-143W were obtained over the range of 1000 to 10,000 psig at 100°F (Reference 4). Generally the viscosity of the PR-143 material was more sensitive to pressure than a mineral oil of comparable V.I. and 100°F viscosity at atmospheric pressure.

Flammability

No autogenous ignition, flash or fire points were found for the PR-143 fluids up to 1000°F. Needless to say, this would be expected based on the structure of these materials. Furthermore, a hot manifold test (not shown in the table) indicated PR-143AB, Lot IV, did not flash or burn after contact with manifold temperatures up to 1240°F (Reference 5).

Volatility

Experiments including constant temperature evaporation, thermogravimetric analysis (TGA) and vapor pressure (isoteniscope) indicate the volatility of the various PR-143 fluids to vary somewhat according to their composition or distribution of molecular weight fractions comprising the materials. Many of the PR-143 materials show generally less volatility than 5P-4E at 550°F and above.

Thermal Stability

According to differential thermal analysis (DTA), the onset of thermal decomposition for PR-143 is above 900°F. Isoteniscope fixes thermal deterioration at 600-650°F as indicated by the development of excess pressure in that region. During the closed-bomb test at 700°F, corrosion of 52-100 bearing steel and naval bronze occurred; although no neutralization number increase or appreciable viscosity change were noted. The 5P-4E experiments also resulted in slight attack on all three metal catalysts.

Hydrolytic Stability

The bulk property change for PR-143 was negligible after the low temperature (200°F) hydrolytic stability test. Little or no weight change of the copper catalyst occurred, but a corroded appearance was noted in one test. The 5P-4E behaved similarly, except the copper specimen did not appear corroded.

Foaming Characteristics

PR-143, Lot I, had very little foaming tendency and a rapid foam collapse at 75°F and 200°F. Another material, PR-143P produced considerable foam and complete collapse was not obtained after 10 minutes at 75°F. 5P-4E had poor foaming characteristics similar to PR-143P.

Engineering Design Properties

The specific heat and thermal conductivity values for PR-143 are lower than those obtained for 5P-4E. Since sufficient heat transfer and thermal diffusion are essential in many high temperature applications, the low thermal conductivity of PR-143 could be of concern unless a system were specifically designed for use with PR-143 fluids.

Bulk modulus data indicate PR-143 to be a relatively compressible fluid, but not to the extent of 5P-4E.

Naturally as a result of the inherent nature of the perfluorinated PR-143 materials their densities would be quite high as was revealed experimentally.

The sonic shear stability of PR-143 was very good when compared with a standard ASTM reference fluid tested for comparison (see footnote in Table 1).

Electrical Properties

Preliminary investigation has shown PR-143 to have good electrical properties for electronic coolant applications. PR-143 was superior to most other fluids evaluated (Reference 6) with respect to volume resistivity, dissipation factor, dielectric constant and electric strength.

Gas Solubility

Air solubility in PR-143W as a function of temperature was measured using gas chromatographic techniques (References 4 and 7).

According to the investigators the PR-143 fluid does not react readily with oxygen up to 400°F. The oxygen and total air solubility curves (not shown) are straight lines indicating increasing solubility over the entire temperature range up to 400°F. Esters and mineral oil neat fluids show a sharp break in the air and oxygen curves at high temperatures indicating that some dissolved oxygen reacts and can no longer be measured as a gas. The PR-143 fluid shows much higher gas solubility values than those measured for mineral oils of the same viscosity level.

In the case of air solubility, dissolved gases are no longer in the same ratio as found in air. The dissolved oxygen represents 28 percent of the total dissolved gas as compared with about 21 percent oxygen in the air used. Gas solubility data with individual gases indicate that oxygen solubility is measurably higher than that of nitrogen at all temperatures studied.

Radiation Stability

Radiation stability of PR-143W was evaluated in the Penn State Nuclear Reactor at 10^7 and 10^8 rads total dose (Reference 4). The fluid was subjected to a mixed dose of gamma radiation as well as thermal and fast neutrons. The property changes for PR-143 after radiation are shown in Table 2. It was a conclusion of the investigators that the data showed good radiation stability for the PR-143 fluid, with gassing and viscosity decrease as evidence of incipient degradation.

Elastomer Compatibility

The results of compatibility tests involving PR-143, Lot II, and various elastomers at 300 to 500°F are given in Table 3. The elastomeric materials were immersed in PR-143 in a beaker maintained at constant temperature in an oven for 168 hours. At 500°F the Vitons have poor compatibility with PR-143 and only the triazine elastomer shows promise. Generally PR-143 did not produce any significant swell of the elastomeric materials in any of the tests. In current applications this would be a problem but it is believed that oversized seals or redesign can circumvent this deficiency.

Additional compatibility studies were made in tests similar to Federal Test Method 3604 to determine the effect of PR-143, Lot III, on the swelling of three O-ring materials, Viton A, Viton B and Silicone LS-53 (Reference 8). The results are presented in Table 4. The tests were made by completely submerging the elastomer in PR-143 contained in a beaker and heated on a hot plate at the desired temperature for 72 hours. The PR-143 caused no swell, but significant shrinkage of the elastomers occurred as in the preceding experiments.

OXIDATION STABILITY AND PR-143-METAL COMPATIBILITY

Oxidation-corrosion (O-C) studies were performed under a variety of conditions on several PR-143 fluids as summarized in Tables 6 through 15. All investigations were made on a micro scale as previously described in detail (Reference 13). In some tests slight modifications were made in test equipment and conditions for the effect on PR-143 stability.

Table 6 summarizes 500 to 800°F O-C results for early PR-143 samples, PR-143M and PR-143P. Similar tests using 5P-4E were made. The PR-143 fluids demonstrated good O-C stability at 600°F; however, above that temperature fluid degradation and serious metal corrosion occurred. Similarly the 5P-4E degraded significantly above 600°F, but no metal corrosion resulted.

O-C data for PR-143, Lot I, evaluated under wet air and wet nitrogen at 700°F, are shown in Table 7. Similar tests were made under dry conditions for comparison. Large negative viscosity changes and much corrosion occurred in wet air and dry air in the presence of metals. Viscosity change and metal corrosion were generally greater in the tests with wet air. In nitrogen atmosphere, wet and dry conditions gave similar results; fluid bulk property change was small and metal corrosion slight compared to the same tests with air.

Table 8 gives metal-compatibility data for PR-143, Lot II, at 700°F with dry air. Those metals with which PR-143 appeared compatible (no fluid degradation and metal corrosion) at 700°F were those with appreciable nickel content (Hastelloy R-235, Inconel, Carpenter 20) and silver.

PR-143, Lot II, was also evaluated in a series of mild corrosion tests at 500 to 700°F to determine the compatibility of the perfluorinated fluid with various metallic and nonmetallic materials in dry air and nitrogen atmosphere (Table 9). Initial tests were made at 500°F in air. Tests resulting in attack on the specimen material or deterioration of the fluid were evaluated again with nitrogen under similar conditions. More specifically, the criteria for good compatibility was that the PR-143 fluid did not degrade and the metal specimens had no more than ± 0.5 mg/cm² weight change with no evidence of corrosion. If the 500°F test in air was

satisfactory, the test was repeated at 600°F. This procedure was followed at 650 and 700°F.

The materials which survived the 700°F tests with air were Carpenter 20 and Inconel 600, both high nickel-content alloys. In nitrogen atmosphere at 700°F only the copper alloys (in addition to the nickel alloys) endured the test.

The effect of various environmental conditions on PR-143 supplemental to those discussed above are shown in Tables 10 through 15 (Reference 14). In addition to compatibility experiments in air and nitrogen with various metals, investigations were carried out to determine the effect of test tube composition and metal specimen cleaning (after the test).

ENGINEERING PERFORMANCE CHARACTERISTICS OF PR-143 FLUIDS

The lubrication capability of PR-143 fluids in four-ball wear, Ryder Gear, rolling contact fatigue and cage compatibility experiments was investigated. Advanced performance characteristics of PR-143 were determined in bearing rig stabilization studies and full scale bearing rig tests.

Wear studies were performed with Shell four-ball wear testers (Reference 11) under various conditions with several PR-143 fluids as shown in Tables 16 through 18. Comparative data are given for 5P-4E in Table 16.

The results in Table 16 indicate the wear scar values for the PR-143 fluids at the lower loads (1,4 and 10 kilograms) were small. At 40 kilogram load the wear scars were generally somewhat larger particularly at 400°F. The appearance of the PR-143 oils after the tests remained unchanged.

The 5P-4E tests resulted in considerably more wear under almost every test condition. In many cases the wear scars after the 5P-4E tests were 3 to 5 times larger than after the corresponding PR-143 tests.

In other wear tests (Table 17), PR-143W appeared to be an effective lubricant over the wide range of conditions employed (Reference 4). The investigators made several observations which may help to define the role of PR-143 in boundary lubrication. The wear scars show less surface roughness or gouging and more evidence of uniform etching or corrosion. The etched or corroded appearance of the scar surface is apparent even in low load, low temperature runs. At the high temperatures, especially at 700°F, the entire ball had an etched appearance and all the balls were discolored.

Additional four-ball investigations were made (Reference 15) at 400 and 600°F with PR-143, Lot V (Table 18). Generally the wear scars produced were slightly larger at the higher temperature (600°F) and speed (1280 RPM). In most tests the 52-100 steel balls had larger wear scars than M-10.

Ryder Gear

Ryder Gear scuff-limited load evaluations were performed on PR-143P and 5P-4E (Reference 16). Duplicate tests on each fluid were run at 400°F bulk oil temperature,

425°F gear temperature using Nitralloy N gear material. The results for the PR-143 fluid are as follows:

<u>Test 1</u>	<u>Test 2</u>
4450 lb/in	4860 lb/in
Average 4660 lb/in	

The results of the two determinations made on 5P-4E are:

<u>Test 1</u>	<u>Test 2</u>
1040 lb/in	1040 lb/in
Average 1040 lb/in	

Although the initial Ryder Gear tests show PR-143 to be far superior to 5P-4E in load-carrying ability, a number of unusual phenomena were observed by the investigators.

In both PR-143 tests, at the failure load and the load before failure, the induction heat was required only to bring the gear to the test temperature of 425°F. After the load was applied at 425°F, the temperature of the test gear increased slowly and uncontrollably. At the load before failure the gear temperature increased to 450°F before the end of the load period, but the increase in scuff was slight. At the failure load the same increase was noted for about three minutes at which time the gear temperature increased rapidly, within seconds, to over 600°F. At this point noise from the machine indicated gear failure and the run was terminated. On inspection of the test gear it was observed that the working face of seventeen teeth was completely destroyed.

In the PR-143 runs the lubricant and test section, including the test oil system, gear cover, and test gears, were found to be exceptionally clean and free of deposits.

The used PR-143 fluid did not darken as did 5P-4E, but was cloudy initially. Upon standing, much of the fluid had cleared. Apparently the cloudiness was due to dissolved or suspended air. A small amount (about 3 ml) of some insoluble black oil was observed on top of the used PR-143 drained from the system. This contaminating oil was believed to have come from the Bourdon pressure gage tube.

After these initial tests, additional work was done with PR-143, Lot I, which resulted in considerably lower Ryder Gear values (Reference 16). In view of this, and the unusual phenomena associated with the aforementioned PR-143P tests, a more extensive Ryder Gear study would have to be made to obtain a meaningful Ryder Gear rating.

Rolling Contact Fatigue

The rolling contact (RC) fatigue tests were performed on a rig developed by General Electric, Advanced Engine Technology Department (Reference 8). The RC rig tests were performed on PR-143, Lot III, under the following test conditions:

Material:	M-50 cons. arc. melted
Test Temp:	room temp, 425°F, 500°F, 600°F

Stress: 700,000 psi max. Hertz
 Test Speed: 25,000 stress cycles/min.
 Lub Feed Rate: 20 drops/min.

Ten RC rig tests were performed on the PR-143 fluid at room temperature and 425°F. One test was made at 500°F and 600°F. The number of stress cycles to failure obtained are given in Table 19. The B-10 and B-50 life at room temperature and 425°F as determined from a statistical distribution curve are also given in the table.

The RC rig results show the PR-143 fluid to have excellent fatigue life at room temperature, 425°F, 500°F and 600°F.

Cage Compatibility

A materials screening program associated with a high-speed, high temperature bearing study was begun in which candidate cage materials were evaluated for wear resistance when lubricated with PR-143, Lot III, at 700°F (Reference 17). The cage materials used, M-1 (R_c 60) steel and S-Monel (R_c 33), were two of the best wear resistant materials to date. The test conditions and resultant data are presented in Table 20.

According to the investigators the M-1 steel was much more wear resistant than the S-Monel material in PR-143 at 700°F. Examination of the cage specimens after the tests indicated that the S-Monel cage specimen had dark wear scars; whereas the scars in the M-1 specimen were clean and exposed the bare metal.

Bearing Stabilization

Bearing stabilization tests were made on PR-143P and 5P-4E (for comparison) under the following conditions, using an Erdco Universal Tester and WADD bearing head: 100 mm bearing size, 10,000 RPM speed, 100 ml/minute oil flow, stabilization at oil-in temperatures of 300, 400, 500, 600 and 700°F within a 40 minute minimum - 120 minute maximum time interval (References 18 and 19).

Prior to the PR-143 stabilization test the bearing rig was flushed first with Freon 113 then with some PR-143 fluid. Before the 5P-4E run the rig was cleaned with Freon 113 and flushed with fresh 5P-4E.

Table 21 summarizes bearing stabilization data for PR-143P, including viscosities. Note the stabilization test made at 300°F (oil-in) was repeated between the 600 and 700°F tests with excellent duplication of results. Examination of the bearing head after the 700°F test revealed the head and bearing to be dry except for the fluid that had drained to the bottom. The bearing and outer race were blue in color and no deposits resulting from liquid degradation were found. The color of the test fluid did not change significantly during testing.

The total volume of PR-143 charged to the bearing rig was 640 ml (500 ml initial charge and 140 ml make-up). A fluid balance was not attempted due to the leakage and evaporation which occurred during the testing.

Table 22 summarizes the 5P-4E bearing stabilization tests, including viscosities. Examination of the bearing head after the 700°F test showed the head and bearing to

have a light fluid film. The hub and outer bearing race were blue-brown in color and no deposits resulting from fluid degradation were observed in the head. Inspection of the test fluid heater showed light sludge deposits on the surface of the heater element (PR-143 gave only a dulling of the aluminum element). The 5P-4E fluid darkened slightly at 500°F and turned dark brown at 600°F and brown-black at 700°F.

The total volume of 5P-4E charged to the bearing rig was 600 ml (no make-up was required) with 25 ml samples taken for viscosity and neutralization number determinations after each stabilization temperature.

In summary, PR-143P and 5P-4E stabilized at all test temperatures within the 120 minute (maximum) limit. Generally in the 5P-4E tests the bearing stabilized 20 to 30°F below the corresponding PR-143 temperatures. Essentially the viscosity changes at 100°F for 5P-4E were negligible, while the viscosity of PR-143 increased 10 to 15 percent. No neutralization number change was detected for 5P-4E.

Full-Scale Bearing Rig

The full-scale bearing rig performance of PR-143, Lot I was investigated under two sets of operating temperatures as outlined in Tables 23 and 24 (Reference 16). A brief summary of the test conditions is given in the tables, together with fluid performance data and deposit demerit ratings. A more detailed description of the bearing rig test equipment, operating procedures and techniques employed have been reported previously (Reference 16).

The first bearing test (Table 23) was conducted at a sump temperature of 650°F and a bearing temperature of 700°F. According to the evaluators the test bearing, when operating with PR-143, had an unusually high stabilization temperature of 490°F. This temperature was approximately 125°F greater than that encountered for all other lubricants evaluated in the past. Thus the usual maximum bearing stabilization temperature of 375°F was waived, and the test was allowed to proceed. The test was interrupted at 7.5 hours and again at 11.7 hours as a result of test oil pressure pump failure. These failures were the result of hard deposits accumulating between the pump shafts and their bearing surfaces. Later, all items located within the oil sump were completely coated with a reddish brown substance similar in appearance to iron oxide. The oil sump wall and bottom were completely covered with a heavy deposit of dark brown material that appeared to be flaking away from the sump. The end cover of the bearing machine was removed and the machine inspected.

In order to arrive at some idea of the relative amount of deposit formation, a deposit rating was obtained. It was necessary to ignore the color of the various deposits and rely on consistency and thickness alone. The largest portion of the deposits was in the sludge or carbon class with considerable flaking. Only a small quantity of deposits, present on the test bearing rollers, appeared to fall into the varnish class. The deposits rated as sludge ranged from greyish-white to dark grey and brown in color and those classed as carbon were light reddish-brown to dark brown. Deposit formation was so severe that, following 11.7 hours of test time, the overall deposit rating was an unusually high 580. Thus at this time the test was stopped.

Analyses of the deposits at various locations in the bearing machine and sump indicated iron to be the major constituent (over 10 percent), together with a substantial amount of chromium (up to 10 percent).

Metal specimens mounted in the sump also showed evidence of serious corrosion. In particular, titanium and stainless steel had significant weight losses. The changes in weight of the metals resulting from exposure to PR-143 at 650°F are given below:

<u>Metal Specimen</u>	<u>Weight Change, mg/cm²</u>
Aluminum	+0.22
Titanium	-1.44
Silver	-0.02
Steel	+2.74
Stainless Steel	-2.24

Although PR-143 had only a 14 percent viscosity increase at 210°F after the test, the investigators (Reference 16) concluded that in view of the excessive deposit formation and corrosive action, PR-143 was considered unsatisfactory in the bearing test at 650°F sump and 700°F bearing temperatures.

A second bearing rig test was made (Table 24) on PR-143 using a sump temperature of 500°F and bearing temperature of 588°F (average temperature over a 48 hour period). Actually a bearing temperature of 550°F was planned but it was not possible to control the bearing at that temperature due to an increase in temperature without external heating during the test.

This second run also had an elevated bearing stabilization temperature, which reached a maximum of 468°F, and again required waiver of the usual stabilization temperature maximum of 375°F. Excluding bearing temperature, the test apparatus performance was satisfactory throughout the run. During the final 16 hours of run time, the test bearing temperature reached a maximum of 605°F.

Periodic inspection of deposit accumulation in the bearing machine showed that the heaviest deposit build-up was during the final 16 hours of operation. Upon completion of the test, the bulk oil was dark brown in color caused by small-particle suspension. The sump wall and bottom were completely coated with a brown substance. A similar deposit, removed by moderate wiping, covered entirely all metal surfaces submerged in the PR-143.

In view of the corrosive behavior experienced in the first test, the test bearing used in the second test was weighed before and after the test with the following results:

	<u>Weight, grams</u>	
	<u>Outer race</u>	<u>Inner race- cage assembly</u>
Before test	547.12	890.52
After test	546.47	889.20
Loss	0.65	1.32

Considerable pitting and corrosion of the inner and outer races was noted; however, the metal specimens in the sump had no significant weight changes. According to the evaluators the apparent excellent oxidative stability of the PR-143 fluid was overshadowed by its high corrosivity, which was not adequately reflected by the overall deposit rating (111.8) since the deposits were mostly not of the usual carbonaceous type, but appeared to be primarily corrosive products.

In brief summary, PR-143, Lot I, did not satisfy the minimum performance requirements at the more strenuous test environment, namely 650°F sump and 700°F test bearing temperatures. Deposit formation, which occurred early in the test, was considered of the severest type. The second bearing test at 500°F sump and 588°F bearing temperatures resulted in an overall deposit formation that compared rating-wise to those obtained with 5P-4E at corresponding temperatures of 700 and 750°F (Reference 16). However, the rate of PR-143 degradation at these temperatures was slight.

As a result of similar tests with 5P-4E the investigators (Reference 16) concluded that the polyphenylether could be expected to give satisfactory performance at 600°F sump and 650°F bearing temperatures, both from the standpoint of deposit formation and oil deterioration.

PR-143 GREASES

Grease compositions prepared from PR-143 fluids and various thickeners were subjected to numerous tests including penetration, evaporation, Pope Spindle and LOX compatibility. The results of the investigation, together with the composition of the greases evaluated, are summarized in Table 25.

Additional data obtained on a Du Pont formulated PR-143 grease (PR-240) are presented in Table 26 (Reference 20).

SECTION III

CONCLUSIONS AND RECOMMENDATIONS

The laboratory investigations and performance data show the PR-143 fluids to be an extremely interesting new class of materials. These fluids offer a considerable improvement over 5P-4E polyphenylether in low temperature fluidity while maintaining outstanding high temperature oxidative stability and good lubricating characteristics. Other properties, such as a high degree of fire resistance and LOX compatibility, make PR-143 a promising candidate for various specialized applications.

Of the short-comings revealed by this investigation, the most serious is the corrosive effect of the fluid on some metals at high temperatures. At 550°F and above, PR-143 fluid corrodes most ferrous alloys and certain titanium alloys. Super alloys having high nickel or cobalt content are not affected by PR-143 at temperatures up to 700°F. It is recommended therefore, that where practical, these super-alloys be selected as materials of construction during the design stage of advance systems in order to capitalize on the good properties of the PR-143 fluids without paying the corrosion penalty.

Another promising route for obtaining a compatible PR-143 fluid-metal system is through the use of additives. Preliminary work currently being done in this area indicates certain PR-143 formulations to have improved metal compatibility characteristics over the base fluid at 550 to 600°F (Reference 21).

The relatively high density of PR-143 may prove to be a deterrent to their use where weight is a severe handicap.

Design considerations, based on a good knowledge of the outstanding properties as well as the serious short-comings of PR-143 materials, should provide workable solutions to some of the fluid and lubricant problems of advanced systems.

SECTION IV

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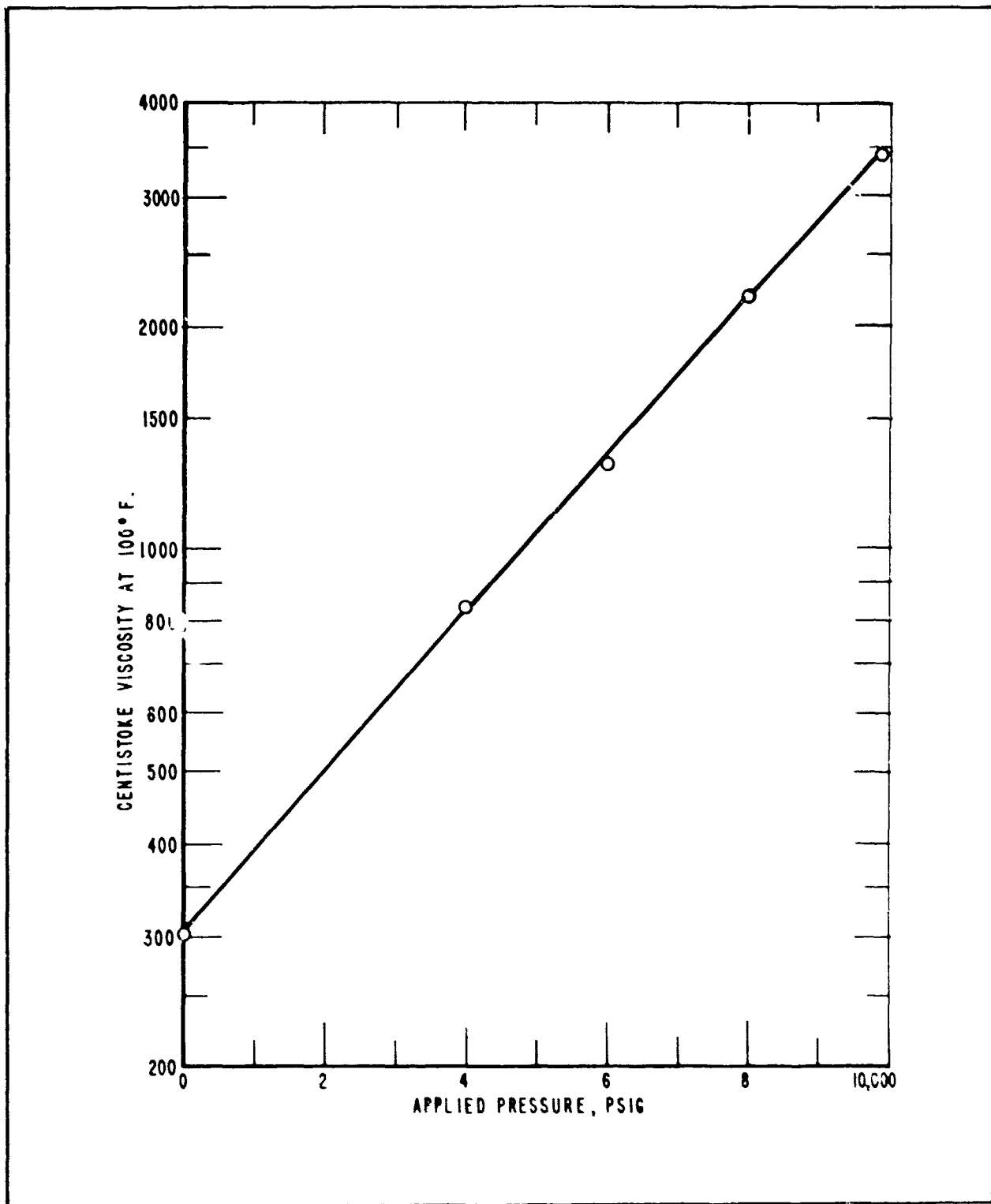


Figure 1. Effect of Pressure on the Viscosity of PR-143W (Tests Conducted in a Pennsylvania Refining Laboratory Pressure Viscometer at 100°F.)

TABLE I
GENERAL PHYSICAL AND CHEMICAL PROPERTIES OF PR-143 FLUIDS

Evaluation	PR-143M	PR-143P	PR-143, Lot I	PR-143, Lot II	PR-143, Lot III	PR-143W	PR-143X	PR-143AB, Lot IV	SP-4E
Viscosity, centistokes @									
-25°F	22,100	24,900	42,000	50,100	55,400	38,600	7,450	20,200	—
0°F	220	219	283	313	335	299	96.1	3,640	365
100°F	23.0	20.4	25.5	27.4	29.0	28.1	11.2	59.6	13.1
210°F	—	—	3.64	3.85	4.11	4.11	1.93	7.95	2.12
400°F	1.96	—	1.90	2.00	2.12	—	—	1.47	1.29
500°F	1.50	—	1.45	1.53	1.66	—	—	—	1.16
550°F	—	—	—	0.79 ^a	—	—	—	—	0.70
700°F	—	—	—	—	—	—	—	—	—
Viscosity index	—	114	115	115	116	119	110	—	-81
ASTM slope	—	0.622	0.609	0.602	0.596	0.584	0.678	—	—
Pour point, °F	-30	-30	-25	-15	-15	-20	-45	—	+40
Autogenous ignition temperature, °F	> 1000	—	—	> 1000 ^b	—	—	—	—	> 1000
Flash point, °F	> 850	—	—	> 940 ^b	—	—	—	—	650
Fire point, °F	> 850	—	—	—	—	—	—	—	695
Evaporation, 6 1/2 hrs. % Wt. loss @ 100°F Visc increase @									
500°F	28.2	10.2	5.8	6.2	1.4	1.9	—	—	5.7
	45.0	—	9.4	9.0	—	—	—	—	1.0
550°F	54.8 ^d	—	11.9	18.1	—	—	—	—	27.6
	188	—	15.2	16.2	—	—	—	—	9.0
600°F	—	45.8	34.8	30.2	—	—	—	—	58.5
	—	58.0	33.6	23.3	—	—	—	—	13.0
Volatility by TGA, % Wt. loss @									
464°F	—	—	0	0	0	—	—	—	—
680°F	—	—	10	7.8	5.5	—	—	—	—
739°F	—	—	25	18.0	16.2	—	—	—	—
792°F	—	—	50	39.6	34.6	—	—	—	—
829°F	—	—	75	65.3	56.0	—	—	—	—
889°F	—	—	100	99.4	93.2	—	—	—	—
Vapor pressure, mm Hg @									
350°F	2.6	0.4	—	0.2	—	—	—	—	0.3
400°F	3.6	1.2	—	0.6	—	—	—	—	0.7
500°F	10.5	9.5	—	3.4	—	—	—	—	3.2
550°F	26.0	25	—	7.7	—	—	—	—	6.8
600°F	58.0	55	—	15.5	—	—	—	—	12
550°F	126	125	—	115	—	—	—	—	22
675°F	—	200	—	250	—	—	—	—	—
700°F	240	600	—	680	—	—	—	—	39
Isoteniscope ¹ initial thermal decomposition temperature, °F	389	650	—	600	—	—	—	—	649
Differential thermal analysis, onset of thermal decomposition, °F	—	910 ^h	—	—	—	—	—	—	> 990 ⁱ

TABLE I (Cont'd)

GENERAL PHYSICAL AND CHEMICAL PROPERTIES OF PR-143 FLUIDS

[illegible]

TABLE I (Contd)
GENERAL PHYSICAL AND CHEMICAL PROPERTIES OF PR-143 FLUIDS

Evaluation	PR-143M	PP-143P	PR-143, Lot I	PR-143, Lot II	PR-143, Lot III	PR-143W	PR-143X	PR-143AB, Lot IV	SP-4E
Bulk modulus, isothermal secant, 3000 psi, $\text{psi} \times 10^{-3}$ @ 100°F	—	—	—	—	—	—	—	—	332
Density, gm cc @ 0°F	—	—	—	—	—	—	—	—	150
75°F	—	—	—	1.9567	—	—	—	—	—
100°F	—	—	—	1.9051	1.9067	—	—	—	—
200°F	—	—	—	1.8838	—	—	—	—	1.1863
300°F	—	—	—	1.7865	—	—	—	—	1.1443
400°F	—	—	—	1.6908	—	—	—	—	1.1033
500°F	—	—	—	1.5963	—	—	—	—	1.0618
Sonic shear ^a , visc change @ 100°F after irradiation, %	—	—	—	+ 0.4'	—	—	—	—	1.0231
Surface tension @ 77°F, dynes/cm	—	—	17.8	17.8	17.8	—	—	—	-2.6 ^s
Dielectric constant @ 1000 cps @ 70°F	—	—	—	2.3	—	—	—	—	47.1
300°F	—	—	—	2.2	—	—	—	—	—
Dissipation factor @ 1000 cps @ 70°F	—	—	—	0.0005	—	—	—	—	—
300°F	—	—	—	< 0.0005	—	—	—	—	—
Volume resistivity, ohm-cm DC @ 70°F	—	—	—	1.0×10^{14}	—	—	—	—	—
300°F	—	—	—	1.3×10^{14}	—	—	—	—	—
Electric strength, plane to sphere with 0.025 in. gap KV @ 70°F	—	—	—	13.0	—	—	—	—	—

- Indications of boiling
- The sample completely evaporated before flash and fire point could be obtained. Maximum temperature attained was 940°F.
 - As per ASTM-D 972
 - Test was made on reclaimed PR-143M
 - Determinations performed in a platinum crucible at atmospheric pressure with 0.25 ft³/hr. helium sweep and heating rate of 6°C/min.
 - Phoenix Chemical Laboratory Isotenscope Method A (Reference 9).
 - Phoenix Chemical Laboratory method with one atmosphere of nitrogen (Reference 10).
 - Exotherm at 910°F suggested a general reaction within the bulk sample.
 - An intense endothermic boiling peak at 990°F
 - Penn State - type thermal stability test at constant temperature (Reference 11).
 - Initially + 0.24. Upon wiping, the bronze catalyst weight change dropped to + 0.06.
 - Slight corrosion
 - Block corrosion
 - As per Federal Test Method Standard NQ 791, Method 3457.
 - As per ASTM-D 892
 - As per method performed at Phoenix Chemical Laboratory (Reference 12).
 - As per MIL-L-23699-WEF
 - Value for ASTM Reference Fluid A run for comparison was -11.4%.
 - Value for ASTM Reference Fluid A run for comparison was -11.1%.

TABLE II

Effect of Mixed Thermal Neutron
and Gamma Radiation on PR-143W

	PR-143W before exposure	PR-143W after 10^7 rads exposure	PR-143W after 10^8 rads exposure
Gaseous product, vol.gas/vol.liquid	0	1	6
Viscosity, cs, @ 100°F	300	276	158
Viscosity change @ 100°F, %	-	-8	-47
Neutralization $N^{\frac{O}{7}}$, mg KOH/g	0.1	2.9	11.6

TABLE III
ELASTOMER COMPATIBILITY OF PR-143, LOT II

Material	Elastomer Identification	Original Properties of Elastomer			Properties of Elastomer After immersion in PR-143, Lot II for 168 hrs. @ 200°F				Properties of Elastomer After immersion in PR-143, Lot II for 168 hrs. @ 300°F				Properties of Elastomer After immersion in PR-143, Lot II for 168 hrs. @ 400°F			
		Tensile	Elongation	Hardness	Tensile Loss, %	Elongation Loss, %	Hardness Change	Volume Change, %	Tensile Loss, %	Elongation Loss, %	Hardness Change	Volume Change, %	Tensile Loss, %	Elongation Loss, %	Hardness Change	Volume Change, %
Fluorosilicone	L563	1550	310	44	28	17	+13	+0.8	39	7	+13	-2	100	81	+4	-2
Ethylacrylate	4021	1850	280	70	0	16	+5	+0.7	—	37	—	0	11	99	+29	-15
Methylsilicone	K1046	990	280	55	17	11	+11	+1.3	26	4	+9	+5	68	55	+15	-4
Viton A	AMS-D	2800	175	85	16	0	+2	+0.8	21	—	+2	-0.5	38	—	+4	-0.8
Viton A	F 34	1995	180	81	—	—	—	—	—	—	—	—	—	—	—	-21.0
Viton A, HV	F 3	1275	110	84	—	—	—	—	—	—	—	—	—	—	—	-17.5
Viton B	F 72	1965	275	76	—	—	—	—	—	—	—	—	—	—	—	-18.0
Triazine	T 23	220	120	58	—	—	—	—	—	—	—	—	—	—	—	-5.7
Urethane	61-19 C	2500	250	90	—	—	+2	+0.9	—	—	—	—	—	—	—	—
Vibronene (urethane)	F 21	3350	160	86	—	—	—	—	—	—	—	—	Brittle	—	—	—
Hydron	B C	4740	160	91	—	31	0	+0.9	54	75	+6	-2	—	—	—	—
Buyl 125	18 D	2460	400	67	15	25	+6	+1	100	—	-32	0	—	—	—	—
VPA	2543	2500	350	80	23	14	0	-3	Brittle	—	—	-5	—	—	—	—
Natural Rubber	2544	985	150	78	68	43	-1	+0.2	—	—	—	—	—	—	—	—
Neoprene, BR T	B B	2330	160	82	2	40	+9	+0.3	Brittle	—	—	-1	—	—	—	—
Hycol 100 (Bufo H)	H Stock	3820	500	67	10	40	+12	0	—	—	—	—	—	—	—	—
Polyisobutylene (Thiokol)	26 B	975	260	66	25	—	+1	-0.8	—	—	—	—	Brittle	—	—	—
Cis-1,4-Polybutadiene	14 B	1720	340	68	58	68	+13	+0.7	—	—	—	—	—	—	—	—
Synzel 1012, SBR	21 E	3220	500	70	49	52	+6	+1.3	—	—	—	—	—	—	—	—
EPT Sulfur Cure	A 87	4360	500	76	33	40	+3	+1.4	—	—	—	—	Brittle	—	—	-6
EPT Peroxide Cure	A 184	4550	740	63	—	5	+5	+1.3	60	47	+14	0	Brittle	—	—	-6
Polysulfoneamide XP 139	341 A	2480	790	55	—	—	—	—	Brittle	—	—	—	—	—	—	-14
Cyanoacryl, Acrylic B Cure	340 A	1460	355	57	—	—	—	—	—	25	+13	+2	—	—	—	—

a. Designed by the Elastomers and Coatings Branch, Air Force Materials Laboratory.

b. Approximate values.

TABLE IV
Elastomer Compatibility of PR-143, Lot III

Material	Test temp., °F	Volume change, %
Viton A	400	-1.18
Viton B	400	-1.14
Silicone LS-53	400	-0.91
Viton A	500	-6.85
Viton B	500	-6.40
Silicone LS-53	500	-5.96

TABLE V
Instrumental and Chemical Analyses of PR-143 Fluids

ANALYSIS	PR-143P	PR-143, Lot I	PR-143, Lot II	PR-143, Lot III	PR-143W
Ultraviolet Spectrum, 10mm cell	Transparent 2100A	above	Transparent 2100A	above	-
Infrared Spectrum	Typical for nonfunctional highly fluorinated compound				-
Emission Spectrum, p.p.m. metals					
Al	0.1-0.5	0.1-0.5	-	-	-
Ca	0.02-0.1	0.02-0.1	-	-	-
Cu	0.05-0.2	0.05-0.2	-	-	-
Fe	0.005-0.02	0.005-0.02	-	-	-
Mg	0.05-0.2	0.02-0.1	-	-	-
Mn	0.02-0.1	0.02-0.1	-	-	-
K	0.05-0.2	0.05-0.2	-	-	-
Si	0.02-0.1	0.02-0.1	-	-	-
Ag	0.002-0.01	0.002-0.01	-	-	-
Na	0.2-1.0	0.2-1.0	-	-	-
Fluoride ion, p.p.m.	-	<1	<1	<1	1.5
Hydrofluoric acid, p.p.m.	-	3	<1	1	1
Neutralization N ^o , mg KOH/g	<0.01	<0.01	<0.01	-	-
Refractive Index, N _D ²³	-	-	1.30226	-	-
Boiling point, °F	-	-	695	-	-

TABLE VI

Oxidation and Oxidation-Corrosion Evaluation of PR-143 Fluids At 500 - 800°F

Fluid	Test temp., °F	Test time, hours	Fluid loss, %	Viscosity (cs) change @ 100°F, %	Viscosity (cs) change @ 210°F, %	Neut. No increase, mg KOH/g	Metal weight change (mg/cm ²) and appearance					Remarks	
							aluminum alloy a.	silver	titanium alloy b.	M-10 tool steel	301 stainless steel		
PR-143M	500	24	38.0	+ 73.6	+ 59.2	0.0	NO METALS USED					No change in appearance of fluid.	
PR-143M	500	24	39.7	+ 80.0	+ 64.3	0.0	NO METALS USED					No change in appearance of fluid.	
PR-143M	500	24	38.3	+ 78.2	+ 62.6	0.0	-0.04	+0.02	+0.02	0.00	-0.04	M.T.	No change in appearance of fluid. c.
PR-143M	500	24	40.0	+ 87.7	+ 69.6	0.0	-0.04	0.00	+0.02	+0.02	-0.04	M.T.	No change in appearance of fluid. c.
^d PR-143M	600	19.5	4.5	+ 7.7	+ 6.1	0.0	0.00	-0.10	-0.28	-0.04	-0.04	L.T.	No change in appearance of fluid.
^{d, f} PR-143M	700	24	20.0	- 8.9	- 5.8	0.0	+0.14	0.00	-0.18	+1.72	+0.28	M.C.	No change in appearance of fluid. A gray-blue film formed on air tube and test tube.
^{d, g, h} PR-143M	800	19.5	49.2	- 77.4	- 67.3	0.0	-0.42	-0.18	-0.14	+1.62	-3.34	H.C.	No change in appearance of fluid. A gray-blue film formed on air tube and test tube.
PR-143P	500	26	15.3	+ 30.6	+ 23.8	0.0	-0.04	-0.06	0.00	-0.02	-0.04	L.T.	No change in appearance of fluid. j.
PR-143P	500	26	18.0	+ 33.3	+ 25.7	0.0	-0.02	-0.04	0.00	0.00	+0.02	L.T.	No change in appearance of fluid. j.
^d PR-143P	600	24	5.2	+ 12.8	+ 10.4	0.0	+0.02	0.00	+0.06	-0.01	0.00	L.T.	No change in appearance of fluid. k.
^d PR-143P	600	24	6.1	+ 15.1	+ 11.7	0.0	+0.01	-0.06	+0.03	0.00	0.00	L.T.	No change in appearance of fluid. k.
^d PR-143P	700	24	41.5	- 56.6	- 45.6	0.0	+0.63	-0.55	-0.16	+0.63	+1.18	H.C.	No change in appearance of fluid. A white film formed on air tube and test tube. Some insolubles produced. l, m
^d PR-143P	700	24	29.8	- 62.1	- 51.0	0.0	+0.56	-0.52	-0.19	+0.45	+1.28	H.C.	No change in appearance of fluid. A white film formed on air tube and test tube. Some insolubles produced. l, m

TABLE VI (Contd)

Oxidation and Oxidation - Corrosion Evaluation of PR-143 Fluids At 500 - 800°F

Fluid	Test temp, °F	Test time, hours	Fluid loss, %	Viscosity (cs) change @ 100°F, %	Viscosity (cs) change @ 210°F, %	Neut. No increase, mg KOH/g	Metal weight change (mg/cm ²) and appearance					Remarks
							aluminum alloy a	silver	titanium alloy b	M-10 tool steel	301 stainless steel	
SP-4E	500	24	2.5	+ 5.5	n.	0.0	NO METALS USED					Fluid slightly darkened. No in-solubles produced.
SP-4E	500	24	2.5	+ 4.4	n.	0.0	NO METALS USED					Fluid slightly darkened. No in-solubles produced.
SP-4E	500	24	5.5	+ 4.9	n.	0.0	-0.02	+0.06	+0.10	+0.06	+0.10	Fluid slightly darkened. No in-solubles produced.
SP-4E	500	24	5.0	+ 5.2	n.	0.0	-0.04	+0.04	+0.02	+0.12	+0.10	Fluid slightly darkened. No in-solubles produced.
^d SP-4E	600	19.5	2.1	+ 6.6	+ 4.6	0.0	-0.02	-0.06	-0.02	-0.02	0.00	Fluid darkened. No in-solubles produced.
^d SP-4E	700	24	8.7	+926	+112	0.0	0.00	-0.06	-0.04	+0.10	-0.08	Fluid darkened badly. No in-solubles produced.
^d SP-4E	800	19.5	25.9	p	p	p	q	-0.06	q	q	q	Fluid charred badly. A solid resin-like material remained.

Micro Oxidation-Corrosion Test Conditions: 20 ml sample, 20-liters per hour air flow rate, five metal specimens, no reflux condenser unless indicated.

Micro Oxidation Test Conditions: Same as O.C test except without metal specimens

- a. Aluminum 2024
b. 6% aluminum - 4% vanadium
c. The 100°F and 210°F viscosities for the overboard distillate collected in a beaker were 78.6 cs and 9.6 cs, respectively.
d. An 18-inch bulb-type reflux condenser was used.
e. Gray-blue deposits on titanium specimen, not removed by wiping.
f. Test sample includes the combined overboard distillate and test tube residue (less some light ends) recovered from 500°F and 600°F tests with metals.
g. Test sample includes the combined overboard distillate and test tube residue (less some heavy ends) recovered from 500°F tests without metals.
h. The test sample refluxed vigorously during test (reflux temperature was about 400°F).
i. Flaking on surface of specimen.
j. The pour point of the combined test tube residues from the duplicate tests was 15°F.
k. The pour point of the combined test tube residues from the duplicate tests was -25°F.
l. Some white polymeric solids precipitated and tiny white crystals were observed near top of test tube.
m. The pour point of the combined test tube residues from the duplicate tests was -40°F.
n. The 210°F viscosity after the test was not determined.
o. Some brown deposits remained on the specimen after wiping.
p. This data was not determined due to the nature of the residue after the test.
q. Heavy, dark deposits remained on specimen after wiping, thus the weight change was not determined.

NT - no tarnish or discoloration
LT - light tarnish or discoloration
MT - moderate tarnish or discoloration
DT - dark tarnish or discoloration
LC - light corrosion
MC - moderate corrosion
HC - heavy corrosion

TABLE VII
COMPATIBILITY OF PR-143, LOT 1 WITH METALS UNDER WET AIR AND NITROGEN AT 700°F

Type of gas	Fluid loss, %	Viscosity (cs) change @ 100°F, %	Neut. No increase, mg KOH/g	Neut. No of distillate, mg KOH/g		Metal weight change (mg/cm ²) and appearance						Remarks
				oil layer	aqueous layer	aluminum alloy a.	silver	titanium alloy b.	M-10 tool steel	301 stainless steel		
DRY AIR	17.9	+18.0	0.0	0.4	—	NO METALS USED						No change in appearance of fluid.
DRY AIR	14.6	+15.9	0.0	0.4	—	NO METALS USED						No change in appearance of fluid.
WET AIR	19.5	+18.4	0.0	d.	81.9	NO METALS USED						No change in appearance of fluid. c.
WET AIR	18.9	+17.3	0.0	d.	78.6	NO METALS USED						No change in appearance of fluid. c.
DRY AIR	54.5	-78.6	0.0	5.7	—	+0.58 D.T.	-0.28 L.C.	-0.52 L.C.	+0.38 L.C.	+0.04 % N.T.	No change in appearance of fluid. f.	
DRY AIR	50.5	-77.3	0.0	6.6	—	+0.56 D.T.	-0.32 L.C.	-0.36 L.C.	-0.62 L.C.	+1.08 H.C.	No change in appearance of fluid. f.	
WET AIR	53.0	-88.5	0.0	d.	>300	+0.13 L.T.	-0.21 L.C.	-1.25 H.C. g.	-2.22 H.C. g.	-2.04 H.C. g.	No change in appearance of fluid. h.	
WET AIR	52.0	-81.9	0.0	d.	>300	-0.21 L.C.	-0.81 H.C. g.	-2.00 H.C. g.	-2.00 H.C. g.	-3.79 H.C. g.	No change in appearance of fluid. h.	
DRY NITROGEN	i	-14.8	0.0	0.3	—	+0.12 L.T.	0.00 N.T.	+0.22 D.T.	-0.04 M.T.	+0.05 L.T.	No change in appearance of fluid. j.	
DRY NITROGEN	13.5	-14.1	0.0	0.7	—	+0.10 L.T.	0.00 N.T.	+0.20 D.T.	+0.11 M.T.	-0.06 L.T.	No change in appearance of fluid. j.	
WET NITROGEN	i	-12.7	0.0	d.	52.9	+0.10 L.T.	0.00 N.T.	+0.77 D.T.	+0.13 D.T.	+0.06 M.T.	No change in appearance of fluid. k.	
WET NITROGEN	13.7	-14.5	0.0	d.	48.4	+0.10 L.T.	0.00 N.T.	+0.67 D.T.	+0.09 D.T.	+0.05 M.T.	No change in appearance of fluid. k.	

Micro Oxidation—Corrosion Test Conditions: 20 ml sample, 20 liters per hour air flow rate, 24 hours duration, 700°F, five metal specimens, modified distilling receiver and 24-inch spiral condenser used

Inerted Micro Corrosion Test Conditions: Same as O.C. test except with 20 liters per hour nitrogen flow rate instead of air.

- a. Aluminum 2024
b. 6% aluminum - 4% vanadium
c. Some white solids at top of test tube and in modified distilling receiver.
d. The neutralization number of the water saturated oil layer was not determined
e. The metal specimen was moved by mistake
f. A white film on test tube and air tube. Tiny white crystals in condenser which were insoluble in Freon 113, but soluble in cold water.
g. Heavy flaking on surface of metal specimen
h. Copious white solids at top of test tube and in modified distilling receiver.
i. The solids were insoluble in Freon 113, hot PR-143, Lot 1, hot conc. HCl, hot conc. NaOH, and hot water, but soluble in cold 50% HF.

- i. Data not available
j. A black film on test tube and air tube.
k. No black film on test tube and air tube.

- N.T. - No tarnish or discoloration
L.T. - light tarnish or discoloration
M.T. - moderate tarnish or discoloration
D.T. - dark tarnish or discoloration
L.C. - light corrosion
H.C. - heavy corrosion

TABLE VIII

Compatibility of PR-143, Lot II With Metals Under Dry Air at 700°F

Metal e.	Fluid loss, %	Viscosity (cs) change @ 100°F, %	Neut. NO increase, mg KOH/g	Metal Wt. change (mg/cm ²) and appearance			Remarks
				specimen nearest air tube orifice	middle specimen	top specimen	
NONE	12.4	+11.5	0.0	NO	METALS	USED	No change in appearance of fluid.
NONE	18.9	+16.6	0.0	NO	METALS	USED	No change in appearance of fluid.
301 stainless steel	7.5	+ 6.7	0.0	+0.50 L.C.	+0.44 L.C.	+0.56 L.C.	No change in appearance of fluid.
301 stainless steel	8.1	+ 8.0	0.0	+0.56 L.C.	+0.68 L.C.	+0.51 L.C.	No change in appearance of fluid.
302 stainless steel	3.3	+ 8.6	0.0	+0.33 H.C. ^a	+1.30 H.C.	+4.13 H.C.	No change in appearance of fluid.
302 stainless steel	3.6	+ 7.7	0.0	+1.30 H.C.	+2.19 H.C.	+2.44 H.C.	No change in appearance of fluid.
304 stainless steel	0.0	+ 8.9	0.0	+0.58 L.C.	+0.34 L.C.	+0.42 L.C.	No change in appearance of fluid.
321 stainless steel	3.4	+ 6.7	0.0	+1.99 H.C.	+2.31 H.C.	+0.84 H.C. ^a	No change in appearance of fluid.
321 stainless steel	3.7	+ 8.9	0.0	+2.17 H.C.	+2.41 H.C.	+2.10 H.C.	No change in appearance of fluid.
347 stainless steel	3.5	+ 7.7	0.0	+1.72 H.C.	+1.48 H.C.	+1.89 H.C.	No change in appearance of fluid.
347 stainless steel	4.8	+ 7.0	0.0	+1.95 H.C.	-11.25 H.C. ^a	+1.89 H.C.	No change in appearance of fluid.
410 stainless steel	7.5	+ 6.1	0.0	-12.67 H.C.	-11.52 H.C.	-11.51 H.C.	No change in appearance of fluid.
410 stainless steel	7.6	+ 8.6	0.0	-12.71 H.C.	-11.38 H.C.	-11.72 H.C.	No change in appearance of fluid.
410 stainless steel	16.0	+ 4.5	0.0	-10.18 H.C.	- 9.15 H.C.	-10.07 H.C.	No change in appearance of fluid.
M-2 tool steel	23.8	+ 6.7	0.0	-19.24 H.C.	-18.86 H.C.	-18.00 H.C.	No change in appearance of fluid.
M-10 tool steel	8.1	+ 6.4	0.0	+0.57 L.C.	+0.65 L.C.	+0.76 L.C.	No change in appearance of fluid.
M-10 tool steel	8.7	+ 8.3	0.0	+0.62 L.C.	+0.63 L.C.	+0.57 L.C.	No change in appearance of fluid.
52100 tool steel	10.1	+ 6.1	0.0	+1.91 H.C.	+2.35 H.C.	+2.21 H.C.	No change in appearance of fluid.
52100 tool steel	8.8	+ 5.1	0.0	+1.42 H.C.	+1.81 H.C.	+1.15 H.C.	No change in appearance of fluid.
1020 mild steel	12.5	+ 5.1	0.0	-14.57 H.C.	-16.50 H.C.	-12.62 H.C.	No change in appearance of fluid.
titanium alloy	94.3	-97.4	0.1	- 0.16 Gray-Brown	-0.06 Gray-Brown	-0.05 Gray-Brown	No change in appearance of fluid. A white film formed on air tube and test tube.

TABLE VIII (Contd)

Compatibility of PR-143, Lot II With Metals Under Dry Air at 700°F

Metal a.	Fluid loss, %	Viscosity (cs) change @ 100°F, %	Neut. NO increase, mg KOH/g	Metal Wt. change (mg/cm ²) and appearance			Remarks
				specimen nearest air tube orifice	middle specimen	top specimen	
b titanium alloy	76.7	-94.0	0.1	-0.04 Gray- Brown	-0.02 Gray- Brown	+0.04 Gray- Brown	No change in appearance of fluid. A white film formed on air tube and test tube.
c aluminum alloy	61.8	-63.6	0.1	+0.13 Dull Gray	+0.19 Dull Gray	+0.10 Dull Gray	No change in appearance of fluid. A white film formed on air tube and test tube.
c aluminum alloy	62.4	-59.4	0.1	+0.17 Dull Gray	+0.13 Dull Gray	+0.12 Dull Gray	No change in appearance of fluid. A white film formed on air tube and test tube.
silver	7.6	+ 7.3	0.0	+0.02 Dull White	+0.05 Dull White	+0.02 Dull White	No change in appearance of fluid.
silver	7.2	+ 7.0	0.0	0.00 Dull White	0.00 Dull White	0.00 Dull White	No change in appearance of fluid.
f, d. brass	10.3	+ 9.6	0.0	+1.41 Dark Brown	+1.41 Dark Brown	+1.53 Dark Brown	No change in appearance of fluid.
f Carpenter 20	11.9	+10.2	0.0	+0.04 L.T.	+0.03 L.T.	0.00 L.T.	No change in appearance of fluid.
Hastelloy R-235	3.7	+ 8.3	0.0	0.00 L.T.	+0.01 L.T.	+0.02 L.T.	No change in appearance of fluid.
Hastelloy R-235	3.5	+ 8.6	0.0	+0.01 L.T.	+0.01 L.T.	+0.03 L.T.	No change in appearance of fluid.
f Inconel 600	8.5	+ 8.0	0.0	+0.22 L.T.	+0.00 L.T.	+0.14 L.T.	No change in appearance of fluid.

Micro Oxidation-Corrosion Test Conditions: 20 ml sample, 20 liters per hour air flow rate, 24 hours duration, 700°F, three identical metal specimens, modified distilling receiver and 24-inch spiral condenser used.

- a. Excessive flaking of metal surface compared to the other metals of the set.
 b. 6% aluminum - 4% vanadium
 Aluminum 2024
 c. 70% copper - 30% zinc
 e. Duplicate tests were made except where indicated
 f. Single test only

L.T. - light tarnish or discoloration
 L.C. - light corrosion
 H.C. - heavy corrosion

TABLE IX

Compatibility of PR-143, Lot II with Metals and Non-metals
under Dry Air and Nitrogen at 500-700°F

a. Metal	Test temp, °F	Gas	Fluid loss, %	Neut. N ^o increase, mgKOH/g	Viscosity (cs) change @ 100°F %	Metal Wt. change, mg/cm ²	Metal appearance
None	500	air	0.0	0.0	-0.6	-	-
None	500	air	0.0	0.0	-0.6	-	-
302 stain. steel	500	air	0.0	0.0	+0.3	0.00	stained
302 stain. steel	500	air	0.5	0.0	+1.0	+0.01	stained
304 stain. steel	500	air	0.9	0.0	+1.6	+0.07	stained
304 stain. steel	500	air	1.2	0.0	+2.1	+0.05	stained
347 stain. steel	500	air	0.3	0.0	0.0	+0.02	stained
347 stain. steel	500	air	0.3	0.0	-0.3	0.00	stained
416 stain. steel	500	air	1.7	0.0	+2.7	+0.11	stained
416 stain. steel	500	air	0.9	0.0	+4.7	+0.11	stained
440 stain. steel	500	air	0.9	0.0	+2.2	+0.07	stained
440 stain. steel	500	air	1.2	0.0	+2.8	+0.06	stained
Carpenter 20	500	air	0.6	0.0	+3.7	+0.12	stained
Carpenter 20	500	air	0.9	0.0	+2.8	+0.12	stained
M-1 tool steel	500	air	0.9	0.0	+2.3	+0.14	stained
M-1 tool steel	500	air	0.9	0.0	+2.0	+0.16	stained heavily
M-2 tool steel	500	air	1.1	0.0	+2.3	+0.84	stained
M-2 tool steel	500	air	1.1	0.0	+2.6	+0.70	heavily stained
M-10 tool steel	500	air	0.3	0.0	+2.0	+0.05	stained
M-10 tool steel	500	air	0.6	0.0	+2.9	+0.10	stained
1020 steel	500	air	0.2	0.0	+0.3	0.00	stained
1020 steel	500	air	0.0	0.0	+0.6	0.00	stained
1040 steel	500	air	0.5	0.0	+2.5	+0.05	stained
1040 steel	500	air	0.3	0.0	+2.6	+0.09	stained
4140 steel	500	air	0.3	0.0	+5.1	+0.12	stained
4140 steel	500	air	0.9	0.0	+2.1	+0.10	stained
Aluminum 2024	500	air	0.0	0.0	+0.3	0.00	no change
Aluminum 2024	500	air	0.0	0.0	+0.3	0.00	no change
Aluminum-bronze (MIL-B-6976)	500	air	0.3	0.0	+1.8	+0.09	stained
Aluminum-bronze (MIL-B-6976)	500	air	0.3	0.0	+3.6	+0.10	stained
Brass (SAE 70A)	500	air	1.7	0.0	+3.9	+0.35	heavily stained
Brass (SAE 70A)	500	air	1.3	0.0	+2.0	+0.51	heavily stained
Copper	500	air	0.0	0.0	-0.3	+0.18	heavily stained
Copper	500	air	0.0	0.0	-0.6	+0.16	heavily stained
Beryllium-copper (QQC-533)	500	air	0.9	0.0	+2.1	+0.20	stained

TABLE IX (Con't)

a. Metal	Test temp, °F	Gas	Fluid loss, %	Neut. N ^o increase, mgKOH/g	Viscosity (cs) change @ 100°F %	Metal Wt. change, mg/cm ²	Metal appearance
Beryllium-copper (QQC-533)	500	air	0.6	0.0	+2.8	+0.12	stained
Titanium alloy (6% Al-4%V)	500	air	0.0	0.0	+1.3	-0.03	stained
Titanium alloy (6% Al-4%V)	500	air	0.0	0.0	+1.0	-0.02	stained
Titanium alloy (4% Al-4% Mn)	500	air	0.9	0.0	+3.0	+0.09	stained
Titanium alloy (4% Al-4% Mn)	500	air	0.6	0.0	+2.0	+0.10	stained
Inconel 600	500	air	0.2	0.0	+1.9	+0.01	stained
Inconel 600	500	air	0.0	0.0	+1.9	0.00	stained
Graphitar	500	air	0.9	0.0	+2.1	-3.50	black deposits
Graphitar	500	air	0.6	0.0	+2.5	-2.83	black deposits
Teflon	500	air	0.6	0.0	+2.9	+2.03	no visible change
Teflon	500	air	0.6	0.0	+1.8	+2.71	no visible change
Buna N	500	air	0.3	0.0	+1.3	-7.44	very brittle
Buna N	500	air	0.6	0.0	+2.9	-7.49	very brittle
Neoprene	500	air	0.9	0.0	+2.2	-15.3	blackened & very brittle
Neoprene	500	air	0.6	0.0	+3.3	-15.7	blackened & very brittle
Viton A	500	air	0.2	0.0	-0.3	-9.29	no visible change
Viton A	500	air	0.2	0.0	+1.3	-9.50	no visible change
None	600	air	0.8	0.0	+0.3	-	-
None	600	air	0.8	0.0	+1.0	-	-
302 stain. steel	600	air	1.0	0.0	+0.3	+0.24	corrosion
302 stain. steel	600	air	0.8	0.0	+1.0	+0.23	corrosion
304 stain. steel	600	air	1.0	0.0	0.0	+0.02	stained
304 stain. steel	600	air	1.0	0.0	+0.3	+0.05	stained
347 stain. steel	600	air	0.8	0.0	+1.6	+0.06	corrosion
347 stain. steel	600	air	0.7	0.0	+1.9	+0.12	corrosion
416 stain. steel	600	air	2.3	0.0	-2.9	-1.81	heavy corrosion
416 stain. steel	600	air	2.3	0.0	-1.9	-1.94	heavy corrosion

TABLE IX (Cont'd)

a. Metal	Test temp, °F	Gas	Fluid loss, %	Neut. N ^o increase, mgKOH/g	Viscosity (cs) change @ 100°F %	Metal Wt. change, mg/cm ²	Metal appearance
440 stain. steel	600	air	0.7	0.0	-3.2	-4.75	heavy corrosion
440 stain. steel	600	air	3.0	0.0	-4.2	-4.70	heavy corrosion
Carpenter 20	600	air	1.0	0.0	+0.6	+0.02	stained
Carpenter 20	600	air	1.0	0.0	-0.3	+0.04	stained
M-1 tool steel	600	air	2.2	0.0	-1.6	-4.04	heavy corrosion
M-1 tool steel	600	air	2.2	0.0	-2.6	-3.81	heavy corrosion
M-2 tool steel	600	air	no data	0.0	-1.3	+11.15	heavy corrosion
M-2 tool steel	600	air	no data	0.0	-1.9	+11.93	heavy corrosion
M-10 tool steel	600	air	1.0	0.0	+0.6	+0.14	stained
M-10 tool steel	600	air	1.0	0.0	+0.6	+0.23	stained
1020 steel	600	air	1.2	0.0	-0.3	+0.98	heavy corrosion
1020 steel	600	air	1.0	0.0	+0.6	-1.64	heavy corrosion
1040 steel	600	air	no data	0.0	-1.6	-7.32	heavy corrosion
1040 steel	600	air	no data	0.0	-1.9	+7.40	heavy corrosion
4140 steel	600	air	2.0	0.0	-2.2	-2.97	heavy corrosion
4140 steel	600	air	1.9	0.0	-1.6	-3.30	heavy corrosion
Aluminum 2024	600	air	1.0	0.0	0.0	+0.05	no visible change
Aluminum 2024	600	air	1.0	0.0	+0.3	+0.06	no visible change
Aluminum-bronze (MIL-B-6976)	600	air	no data	0.0	+1.3	+0.15	stained
Aluminum-bronze (MIL-B-6976)	600	air	no data	0.0	+0.6	+0.16	stained
Brass (SAE 70A)	600	air	no data	0.0	+0.6	+0.69	heavily stained
Brass (SAE 70A)	600	air	no data	0.0	+0.3	+0.97	heavily stained
Copper	600	air	0.8	0.0	+1.9	+0.19	black deposits
Copper	600	air	0.7	0.0	+1.9	-0.22	black deposits,
Beryllium-copper (QQC-533)	600	air	0.9	0.0	-0.3	-0.03	corrosion stained

TABLE IX (Cont'd)

a. Metal	Test temp, °F	Gas	Fluid loss, %	Neut. No. increase, mgKOH/g	Viscosity (cs) change @ 100°F %	Metal Wt. change, mg/cm ²	Metal appearance
Beryllium-copper (QQC-533)	600	air	0.8	0.0	+1.3	+0.04	stained
Titanium alloy (6% Al-4%V)	600	air	7.0	0.0	-25.9	+0.07	dull gray
Titanium alloy (6% Al-4%V)	600	air	9.2	0.0	-28.1	+0.09	dull gray
Titanium alloy (4% Al-4% Mn)	600	air	43.4	0.0	-84.9	-5.88	corrosion
Titanium alloy (4% Al-4% Mn)	600	air	40.2	0.0	-85.6	-6.38	corrosion
Inconel 600	600	air	1.0	0.0	+0.6	+0.03	stained
Inconel 600	600	air	0.8	0.0	+1.0	+0.02	stained
304 stain. steel	650	air	2.0	0.0	0.0	+1.57	heavy corrosion
304 stain. steel	650	air	2.0	0.0	-0.3	+2.90	heavy corrosion
Carpenter 20	650	air	1.5	0.0	+0.3	+0.07	stained
Carpenter 20	650	air	1.5	0.0	+0.3	+0.08	stained
M-10 tool steel	650	air	1.5	0.0	+1.9	+0.79	dull gray
M-10 tool steel	650	air	1.5	0.0	+1.3	+0.76	dull gray
Aluminum 2024	650	air	3.3	0.0	-5.4	+0.12	no visible change
Aluminum 2024	650	air	2.0	0.0	-1.9	+0.10	no visible change
Aluminum-bronze (MIL-B-6976)	650	air	1.7	0.0	0.0	-2.58	corrosion
Aluminum-bronze (MIL-B-6976)	650	air	1.6	0.0	+0.6	+3.42	corrosion
Beryllium-copper (QQC-533)	650	air	1.4	0.0	0.0	0.00	spotted
Beryllium-copper (QQC-533)	650	air	1.5	0.0	-0.6	-0.20	spotted
Inconel 600	650	air	1.3	0.0	-0.6	+0.04	stained
Inconel 600	650	air	1.2	0.0	-0.6	+0.04	stained
Carpenter 20	700	air	3.5	0.0	-0.6	+0.34	stained
Carpenter 20	700	air	3.8	0.0	-0.3	+0.44	stained
Aluminum 2024	700	air	7.1	1.0	-10.2	+0.22	grayed
Aluminum 2024	700	air	16.3	0.6	-38.7	+0.56	grayed
Beryllium-copper (QQC-533)	700	air	4.0	0.0	-1.6	+1.80	black spotted
Beryllium-copper (QQC-533)	700	air	4.2	0.0	-2.2	+0.60	black spotted
Inconel 600	700	air	3.5	0.6	-1.9	+0.10	stained
Inconel 600	700	air	3.6	0.6	-0.3	+0.18	stained

TABLE IX (Cont'd)

a. Metal	Test temp, °F	Gas	Fluid loss, %	Neut. No. increase, mgKOH/g	Viscosity (cs) change @ 100°F %	Metal Wt. change, mg/cm ²	Metal appearance
M-2 tool steel	500	N ₂	0.5	0.0	+0.6	+0.03	stained
M-2 tool steel	500	N ₂	0.5	0.0	+0.6	+0.09	stained
Aluminum 2024	500	N ₂	0.2	0.0	-1.0	-0.04	no visible change
Aluminum 2024	500	N ₂	0.2	0.0	-1.0	-0.04	no visible change
Brass (SAE 70A)	500	N ₂	0.5	0.0	+1.6	+0.01	stained
Brass (SAE 70A)	500	N ₂	0.5	0.0	+1.6	+0.01	stained
Copper	500	N ₂	0.0	0.0	-1.0	-0.03	stained
Copper	500	N ₂	0.0	0.0	-1.0	-0.08	stained
Graphitar	500	N ₂	0.5	0.0	0.0	-2.63	no visible change
Graphitar	500	N ₂	0.2	0.0	-0.3	-1.60	no visible change
Buna N	500	N ₂	0.2	0.0	0.0	-6.75	very brittle
Buna N	500	N ₂	0.2	0.0	+0.3	-6.12	very brittle
Neoprene	500	N ₂	0.2	0.0	-0.3	-9.04	swelled & charred
Neoprene	500	N ₂	no data	no data	no data	-13.52	swelled & charred
Viton A	500	N ₂	0.5	0.0	0.0	-10.65	no visible change
Viton A	500	N ₂	0.5	0.0	+1.6	-10.00	no visible change
302 stain. steel	600	N ₂	no data	no data	no data	+0.07	heavily stained
302 stain. steel	600	N ₂	0.5	0.0	-0.3	0.00	heavily stained
347 stain. steel	600	N ₂	0.2	0.0	+1.0	-0.28	corrosion
347 stain. steel	600	N ₂	0.2	0.0	+0.3	-0.02	corrosion
416 stain. steel	600	N ₂	0.7	0.0	+1.9	-0.04	stained
416 stain. steel	600	N ₂	0.7	0.0	+0.3	-0.05	stained
440 stain. steel	600	N ₂	0.5	0.0	-0.6	+0.14	stained
440 stain. steel	600	N ₂	0.5	0.0	-0.6	+0.11	stained
M-1 tool steel	600	N ₂	0.5	0.0	+1.0	+0.31	stained
M-1 tool steel	600	N ₂	0.5	0.0	+1.0	+0.25	stained
M-2 tool steel	600	N ₂	0.5	0.0	+0.3	+0.37	stained
M-2 tool steel	600	N ₂	0.5	0.0	+0.3	+0.22	stained
1020 steel	600	N ₂	0.2	0.0	-0.6	+0.27	corrosion
1020 steel	600	N ₂	0.2	0.0	+0.6	+0.31	corrosion
1040 steel	600	N ₂	0.5	0.0	+0.3	+0.18	stained
1040 steel	600	N ₂	0.5	0.0	+0.6	+0.17	stained
4140 steel	600	N ₂	0.5	0.0	-1.0	+0.26	stained
4140 steel	600	N ₂	0.5	0.0	-0.3	+0.48	stained

TABLE IX (Cont'd)

a. Metal	Test temp, °F	Gas	Fluid loss, %	Neut. N ₂ increase, mgKOH/g	Viscosity (cs) change @ 100°F %	Metal Wt. change, mg/cm ²	Metal appearance
Aluminum 2024	600	N ₂	0.5	0.0	0.0	0.00	stained
Aluminum 2024	600	N ₂	0.5	0.0	-1.0	0.00	stained
Brass (SAE 70A)	600	N ₂	0.7	0.0	+1.3	+0.13	stained
Brass (SAE 70A)	600	N ₂	0.5	0.0	0.0	+0.12	stained
Copper	600	N ₂	0.2	0.0	+1.0	0.00	stained
Copper	600	N ₂	0.2	0.0	+0.6	+0.02	stained
Titanium (6%Al-4%V)	600	N ₂	0.2	0.0	+0.6	+0.11	blackened
Titanium (6%Al-4%V)	600	N ₂	0.2	0.0	+1.3	+0.15	blackened
Titanium (4%Al-4%Mn)	600	N ₂	0.5	0.0	+1.6	+0.10	stained
Titanium (4%Al-4%Mn)	600	N ₂	0.5	0.0	+0.6	+0.11	stained
302 stain. steel	650	N ₂	0.9	0.0	0.0	+0.10	stained
302 stain. steel	650	N ₂	0.7	0.0	-0.6	+0.11	stained
304 stain. steel	650	N ₂	0.8	0.0	0.0	-0.03	corrosion
304 stain. steel	650	N ₂	1.0	0.0	0.0	-0.02	corrosion
416 stain. steel	650	N ₂	1.0	0.0	-0.3	-0.45	heavily stained
416 stain. steel	650	N ₂	0.7	0.0	-1.3	+0.08	stained
440 stain. steel ^b	650	N ₂	0.7	0.0	-0.3	+0.20	heavily stained
M-10 tool steel	650	N ₂	0.9	0.0	0.0	+0.27	stained
M-10 tool steel	650	N ₂	0.7	0.0	-0.3	+0.29	stained
1040 steel	650	N ₂	1.0	0.0	+1.3	+0.77	corrosion
1040 steel	650	N ₂	1.0	0.0	+0.3	+0.36	corrosion
Aluminum-bronze (MIL-B-6976)	650	N ₂	0.8	0.0	+0.3	+0.14	stained
Aluminum-bronze (MIL-B-6976)	650	N ₂	0.7	0.0	-1.0	+0.17	stained
Brass (SAE 70A)	650	N ₂	1.0	0.0	+0.6	+0.25	corrosion
Brass (SAE 70A)	650	N ₂	1.0	0.0	+0.3	+0.16	corrosion
Copper	650	N ₂	1.0	0.0	+0.3	-0.02	stained
Copper	650	N ₂	1.0	0.0	-0.3	-0.02	stained
Titanium (6%Al-4%V)	650	N ₂	0.8	0.0	0.0	+0.23	corrosion
Titanium (6%Al-4%V)	650	N ₂	0.7	0.0	-0.3	+0.30	corrosion
302 stain. steel	700	N ₂	2.3	0.0	-1.3	+0.26	corrosion
302 stain. steel	700	N ₂	2.6	0.0	-1.6	+0.37	corrosion
Carpenter 20	700	N ₂	2.4	0.0	-1.6	+0.15	heavily stained

TABLE IX (Cont'd)

a. Metal	Test temp, °F	Gas	Fluid loss, %	Neut. N ^o increase, mgKOH/g	Viscosity (cs) change @ 100°F %	Metal Wt. change, mg/cm ²	Metal appearance
Carpenter 20	700	N ₂	2.3	0.0	-1.9	+0.13	heavily stained
Aluminum 2024	700	N ₂	2.2	0.0	-1.9	+0.62	heavily stained
Aluminum 2024	700	N ₂	3.4	0.0	-1.6	+0.47	heavily stained
Aluminum-bronze (MIL-B-6976)	700	N ₂	2.3	0.0	-1.9	+0.25	stained
Aluminum-bronze (MIL-B-6976)	700	N ₂	2.4	0.0	-1.0	+0.12	stained
Copper	700	N ₂	2.3	0.0	-1.9	-0.01	stained
Copper	700	N ₂	2.2	0.0	-1.6	0.00	stained
Beryllium-copper (QQC-533)	700	N ₂	2.2	0.0	0.0	-0.01	stained
Beryllium-copper (QQC-533)	700	N ₂	2.3	0.0	-0.6	-0.04	stained

Micro Test Conditions: 20 ml sample, one-liter per hour gas flow rate, 72 hour duration, one metal specimen, 12-inch bulb-type reflux condenser used.

a. Duplicate tests were made except where indicated

b. Single test only

TABLE X

Effect of Temperature on PR-143^a - Metal Systems

	Weight Change (mg./cm. ² /day)				
	500°F	550°F	600°F	650°F	700°F
<u>Nickel Alloys</u>					
Ni	-0.04	0.00		0.00	-0.05
Inconel	-0.06	0.00	0.00	0.00	0.00
Inconel X		0.00	0.00	+0.02	0.00
Hastelloy C				-0.08 ^b	
Hastelloy X		0.00	+0.03	+0.01	+0.03
Monel				-0.13 ^b	
René 41		0.00	-0.04	-0.01	0.00
<u>Cobalt Alloys</u>					
Stellite 25		0.00		+0.01	+0.02
L-605			-0.05	0.00	
<u>Steels</u>					
1020				-3.25 ^b	
QQ S 636	-0.24	+0.25	+1.26	+1.46	+2.00
<u>Alloy Steels</u>					
SAE 4340		+0.20	+1.55	+2.25	+8.4
52100		+0.14	+1.62	-13.3	-31.0
<u>Stainless Steels</u>					
Stellite N-155		-0.03	+0.03	+0.01	0.00
301	-0.12	0.00	+0.08	+0.01	+1.01
304		-0.01	+0.03	+0.04	+0.94
316		0.00	+0.04	+0.04	+0.53
321		-0.03	+0.01	+0.03	+6.6
405		0.00	+0.23	-0.89	-7.0
410		0.00	+0.06	-3.08	-9.7
440C		+0.04	-0.20	-3.46	-10.9
446		0.00	+0.03	-0.06	-1.68
<u>Nonferrous Metals</u>					
Mg		-0.04	-0.06	+0.05	+0.06
Al		+0.02	+0.07	+0.18	+0.61
Ag	+0.02	-0.13	-0.14	-0.28	-0.58
Ti	0.0	-0.18	-0.73	-1.08	-0.73
Cu		-0.20	-0.77	-0.42	-0.71
Bearing Bronze (85:5:5:5)		+0.12	+0.29	+0.37	+0.88

Micro Oxidation-Corrosion Test Conditions: 20 ml sample in Inconel test tube, 20-liter per hour air flow rate, test durations of 24 and 48 hours, no reflux condenser used. Metal specimens were rinsed with Freon 113 and wiped lightly after test except where noted.

a. The tests were performed on PR-143, Lot I.

b. Metal specimens were rinsed in Freon 113, wiped, then rubbed with scouring powder moistened with water. The cleaning mixture was rinsed away with water. The specimen was dried by dipping in acetone.

TABLE XI

Effect of Gas Composition on PR-143^a - Metal Systems

Material	Weight Change (mg./cm. ² /day)					
	600°F		650°F ^b		700°F	
	Dry Air	Dry Argon	Dry Air	Dry Argon	Dry Air	No Purge Gas
Inconel			-0.16	-0.15		
QQ S 636 Steel	+1.30	+0.01			+2.0	+0.60
1020 Steel			-3.25	-0.07		+0.58
301 Stainless	+0.08	+0.10				
304 Stainless			-1.10	-0.21		
316 Stainless			+0.55	-0.17		
446 Stainless			-0.40	-0.05		
Ti	-1.10	+0.13			-0.73	-0.26
Al					+0.61	+0.42
Ag					-0.58	+0.02
Mg					+0.06	+0.04
Cu					-0.71	+0.08

Micro Oxidation-Corrosion Test Conditions: 20 ml sample in Incone! test tube, 20-liter per hour gas flow rate, test durations of 24 and 48 hours, no reflux condenser used. Metal specimens were rinsed with Freon 113 and wiped lightly after test except where noted.

a. The tests were performed on PR-143, Lot I.

b. Metal specimens were given scouring powder treatment. See footnote b, Table 10.

TABLE XII
^a
 Effect of Moisture on PR-143 - Metal Systems

	Weight Change (mg./cm. ² /day)		
	650°F		700°F
	Dry Air	Humid Air ^b	Humid Air ^c
<u>Nonferrous Metals</u>			
Mg	+0.05	+0.05	
Al	+0.18	-0.03	
Ag	-0.28	-0.07	
Ti	-1.08	+0.46	
Cr		-0.05 ^{c,d}	
Mo			-6.56 ^d
Cu	-0.42	+18	
Bearing Bronze (85:5:5:5)	+0.37	+0.48	

Micro Oxidation - Corrosion Test Conditions: 20 ml sample in Inconel test tube (except where noted), 20-liter per hour air flow rate, test durations of 24 and 48 hours; no reflux condenser used. Metal specimens were rinsed in Freon 113 and wiped lightly after test except where noted.

- a. The tests were performed on PR-143, Lot I.
- b. Air was humidified by passage through water bubblers at 77°F.
- c. Glass test tube used.
- d. Metal specimens were given scouring powder treatment. See footnote b, Table 10.

TABLE XIII

Effect of Air Flow Rate on PR-143^a - Metal Systems

Material	Weight Change (mg./cm ² /day) at 650°F.		
	2 l./hr.	5 l./hr.	20 l./hr.
Hastelloy X	0.00	+0.02	+0.02
QQ S 636 Steel	+1.40	+2.30	+1.50
SAE 4340 Steel	-0.17 ^b	+1.80	+4.30
301 Stainless Steel	+0.60	+0.70	+0.01
405 Stainless Steel	-2.60	-3.00	-1.70
410 Stainless Steel	-4.40	-5.00	-3.10
440C Stainless Steel	-5.90	-7.00	-3.50
446 Stainless Steel	-0.63	-0.71	-0.06
Ti	-1.05	-1.15	-1.30
Cu	-0.49	-0.44	-0.42
Ag	-0.40	-0.19	-0.23
Bearing Bronze (85:5:5:5)	+0.38	+0.48	+0.38

Micro Oxidation-Corrosion Test Conditions: 20 ml sample in Inconel test tube, test durations of 24 and 48 hours, no reflux condenser used. Metal specimens were rinsed in Freon 113 and wiped lightly after the test.

a. The tests were performed on PR-143, Lot I.

b. Error suspected.

TABLE XIV

Effect of Test Tube Composition on PR-143^a - Metal Systems

Material	Weight Change (mg./cm. ² /day) at 700 °F	
	Glass Test Tube ^b	Inconel Test Tube
Inconel	+0.12	+0.08
QQ S 636 Steel	+2.62	+3.63
301 Stainless Steel	+3.96	+7.51
Ti	-2.37	-2.66
Al	+0.44	+0.70
Ag	-0.42	-0.78
Ni	+0.02	+0.04
Mg	+0.20	+0.24
Cu	-0.68	-0.78

Micro Oxidation-Corrosion Test Conditions: 20 ml sample, test durations of 24 and 48 hours, 16-inch pyrex condenser used in all tests. Metal specimens were rinsed in Freon 113 and wiped lightly after the test.

a. The tests were performed on PR-143, Lot I.

b. After each test the glass tube was heavily coated with a white deposit. Analysis of the deposit: F, 50.0%; Na, 12.0%; Al, 9.5%; Si, 1.1%; E, 1.2%.

TABLE XV

Effect of Specimen Cleaning on Weight Change of Metals in PR-143^a

	Wt. Change (mg./cm. ² /day) at 650°F		
	Light Cleaning ^b	Scouring Powder Cleaning ^c	Δ (mg.)
<u>Nickel Alloys</u>			
Ni	0.50	-0.10	0.10
Inconel	+0.02	-0.12	0.14
Inconel X	+0.04	-0.09	0.13
Hastelloy X	+0.03	-0.16	0.19
René 41	0.00	-0.12	0.12
<u>Cobalt Alloy</u>			
Stellite 25	+0.01	-0.14	0.15
<u>Steel</u>			
QQ S 636	+1.11	+0.95	0.16
<u>Alloy Steels</u>			
4340	+4.23	+3.93	0.30
52100	-13.3	-14.3	1.0
<u>Stainless Steels</u>			
Stellite N-15 ^e	0.00	-0.12	0.12
301	+0.05	-0.18	0.23
304	+0.06	-0.05	0.11
316	+0.04	-0.10	0.14
321	+0.03	-0.13	0.16
405	-0.07 ^d	-1.09	1.02
410	-2.51	-2.54	0.53
440C	-2.43	-3.01	0.58
446	+0.03	-0.23	0.26
<u>Nonferrous Metals</u>			
Mg	+0.06	-0.03	0.09
Al	+0.13	-0.07	0.20
Ag	-0.21	-0.58	0.37
Ti	-0.56	-1.00	0.44
Cu	-0.39	-0.80	0.41
Bearing Bronze (85:5:5:5)	+0.36	-0.18	0.54

Micro Oxidation-Corrosion Test Conditions: 20 ml sample in inconel test tube, 20-liter per hour air flow rate, test durations of 24 and 48 hours, no reflux condenser used.

a. The tests were performed on PR-143, Lot I.

b. The specimens were rinsed with Freon 113 and then wiped with glass wool.

c. The specimens were given scouring powder treatment as described in footnote b, Table 10.

d. Error suspected.

TABLE XVI

Four-Ball Wear Characteristics of PR-143 Fluids at 167°F and 400°F

Shell Four-Ball Wear Test Conditions, Average Wear Scar Diameter, mm	PR-143P	PR-143, Lot II	5P-4E
600 RPM, 167°F, 2 hours, 52100 balls @			
1 kg load	-	0.153	0.717
4 kg load	-	0.286	-
10 kg load	-	0.453	1.188
40 kg load	-	0.643	2.079
600 RPM, 400°F, 2 hours, 52100 balls @			
1 kg load	-	0.168	0.420
4 kg load	-	0.208	-
10 kg load	0.307	0.304	0.992
40 kg load	1.011	0.752	1.166
600 RPM, 400°F, 2 hours, M-10 balls @			
1 kg load	-	0.176	0.597
4 kg load	-	0.184	-
10 kg load	0.256	0.230	1.552
40 kg load	0.543	0.680	2.037
1200 RPM, 167°F, 2 hours, 52100 balls @			
1 kg load	-	0.308	0.273
4 kg load	-	0.286	-
10 kg load	0.249	0.452	1.578
40 kg load	0.759	0.782	2.282
1200 RPM, 167°F, 2 hours, M-10 balls @			
10 kg load	0.251	-	2.362
40 kg load	0.459	0.537	3.592
1200 RPM, 400°F, 2 hours, 52100 balls @			
1 kg load	-	0.197	0.462
4 kg load	-	0.225	-
10 kg load	-	0.520	1.046
40 kg load	1.021 ^a	1.050	1.603
1200 RPM, 400°F, 2 hours, M-10 balls @			
1 kg load	-	0.216	0.742
4 kg load	-	0.222	-
10 kg load	-	0.344	1.896
40 kg load	-	0.719	3.732

a. Reclaimed PR-143M

TABLE XVII

Four-Ball Wear Characteristics of PR-143W
Under Controlled Atmosphere

Test temp., °F	Bearing metal, steel type	Atmosphere ^a	Avg. wear scar diam., mm		
			1 Kg	10 Kg	40 Kg
167	52-100	air	0.19	0.28	0.63
400	52-100	0.7 liter air/hr	--	0.34	1.43
400	52-100	0.5 liter N ₂ /hr	--	0.73	1.54
400	M-10	0.7 liter air/hr	--	0.31	0.63
400	440C stainless	0.7 liter air/hr	--	0.38	0.71
500	M-10	0.7 liter air/hr	--	0.45	--
600	M-10	0.7 liter air/hr	--	0.51	--
700	M-10	0.7 liter air/hr	--	0.52	0.70
700	M-10	0.5 liter N ₂ /hr	--	0.54	0.76
700	440C stainless	0.7 liter air/hr	--	0.64	1.32

Four-ball Test Conditions: One hour, 620 RPM; temperature and bearing material as indicated.

a. At test temperatures above 167°F, atmosphere was controlled by introducing air or nitrogen at the rates indicated over the surface of the fluid.

TABLE XVIII

Four-Ball Wear Characteristics of
PR-143, Lot V at 400°F and 600°F

Shell Four-Ball Wear Test Conditions, average wear scar diameter, mm	52-100 bearing material	M-10 bearing material
620 RPM, 400°F, 2 hours, @ 10 kg load	0.30 ^a	0.23 ^a
20 kg load	0.57	0.50
30 kg load	0.63	0.60
40 kg load	0.76	0.50
620 RPM, 600°F, 2 hours, @ 10 kg load	0.66	0.53
20 kg load	0.95	0.82
30 kg load	1.02	0.88
40 kg load	1.08	0.82
1,280 RPM, 400°F, 2 hours, @ 10 kg load	0.52 ^b	0.34 ^b
20 kg load	0.49	0.55
30 kg load	0.83	0.74
40 kg load	0.98	0.95
1,280 RPM, 600°F, 2 hours, @ 10 kg load	0.68	0.62
20 kg load	0.90	0.99
30 kg load	1.06	1.09
40 kg load	1.15	1.24

a. The test was performed on PR-143, Lot II at 600 RPM

b. The test was performed on PR-143, Lot II at 1200 RPM

TABLE XIX

Rolling Contact Fatigue Evaluation of PR-143, Lot III

Test N ^o	Stress cycles to failure			
	room temp.	425°F	500°F	600°F
1	7,495,000	21,075,200	12,631,400	11,095,000
2	17,373,000	11,778,600		
3	16,934,000	14,445,600		
4	19,339,400	11,289,000		
5	11,518,800	15,459,600		
6	1,797,000	7,189,800		
7	7,917,000	8,167,400		
8	10,855,200	18,943,200		
9	2,100,800	13,306,000		
10	9,542,000	11,483,200		

Temperature	Life stress cycle	
	B-10	B-50
room temp	3.3×10^6	10.1×10^6
425°F	7.2×10^6	13.5×10^6

TABLE XX

Cage Compatibility Evaluation of PR-143, Lot III

Cage Material	Lub flow, cc/min.	Temp., °F	Time, hrs.	Average wear scar			N ₂ flow, SCFH	Oil after test		
				Major axis (mm)	Minor axis (mm)	Area (mm ²)		Visc. change @100°F, %	Neut N ^o	Solids, mg/100 ml
M-1 (Rc60)	2.5	700	0.5	2.1	0.8	1.4	2.0	31.1	<0.1	12
			1.5	2.8	1.0	2.1				
S-Monel (Rc33)	3.5	700	0.5	4.8	1.8	5.8	2.0	30.2	<0.1	17
			1.5	6.2	2.4	11.6				

Test Conditions: 1200 RPM, 1000 lb load

TABLE XXI

Bearing Stabilization Evaluation of PR-143P

Fluid in temp, °F	Minutes to Stabilization	Bearing stabilization temperature, °F		
		N ^o 1	N ^o 2	N ^o 3
300	90	474	465	484
400	10	484	481	496
500	40	488	492	500
600	30	513	524	525
300	80	474	465	482
700	30	536	552	550

Total run time under load - 11 hours and 20 minutes

PR-143P Viscosities

Fluid in temp., °F	Viscosity @ 100°F, cs
new	217
300	243
600	247
700	251

TABLE XXII

Bearing Stabilization Evaluation of 5P-4E

Fluid in temp, °F	Minutes to stabilization	Bearing stabilization temperature, °F		
		N ^o 1	N ^o 2	N ^o 3
300	70	436	424	440
400	10	452	446	459
500	10	470	468	481
600	10	490	495	504
700	20	516	530	532

Total run time under load - 5 hours and 20 minutes

5P-4E Viscosities and Neutralization Numbers

Fluid in temp., °F	Viscosity @ 100°F, cs	Neut N ^o , mg KOH/g
new	364	0.0
300	362	0.0
400	363	0.0
500	364	0.0
600	364	0.0
700	367	0.0

TABLE XXIII

Full-Scale Bearing Rig Evaluation of
PR-143, Lot I (Test 1)Demerit Rating

Item	Rating	Factor	Demerits
End Cover	27	1	27
Spacer & Nut	56.5	2	113
Heater Mount (F)	99.5	3	298.5
Heater Mount (R)	94.5	3	283.5
Seal Plate	9	1	9
Test Bearing	550	5	2750
			<u>3481</u>

Overall Rating: $3481/6 = 580.2$

Not included in official rating: Sump - 100% L flaked carbon

Oil consumption rate: 148 ml/hr

Total accumulated filter wt.: Pressure 18g, Scavenge 1.6g

Test Oil Performance

Test Time, hr	Vis, cs at 210°F	% Vis Increase at 210°F	Vis, cs at 100°F
0	25.69	--	286.9
4	27.56	7.2	322.6
8	28.34	10.3	328.6
11.6*	29.16	13.5	343.5

Rig No. 2 with 5 metal specimens in test oil sump

Sump temp, °F 650

Oil in temp, °F 632

Bearing temp, °F 700

Air flow to bearing machining, cfm 0.35

Air Flow to test oil sump, cfm 0

*Deposit formation necessitated test termination

TABLE XXIV
Full-Scale Bearing Rig Evaluation of
PR-143, Lot I (Test 2)

Demerit Rating

Item	Rating	Factor	Demerits
End Cover	18	1	18
Spacer & Nut	85.5	2	171
Heater Mount (F)	57	3	171
Heater Mount (R)	12	3	36
Seal Plate	48	1	48
Test Bearing	45.4	5	227
			<u>671</u>

Overall Rating: $671/6 = 111.8$

Not included in official rating: Sump - 100% L sludge
Oil consumption rate: 15 ml/hr
Total accumulated filter wt.: Pressure 2.6g, Scavenge 1.5g

Test Oil Performance

Test Time, hr	Vis, cs at 210°F	% Vis Increase at 210°F	Vis, cs at 100°F
0	25.69	--	286.9
4	26.01	2.6	296.0
8	26.47	2.9	303.8
12	26.71	3.8	306.6
16	26.82	4.2	308.2
20	26.67	3.7	308.3
24	26.78	4.1	310.6
28	26.94	4.9	312.6
32	27.01	5.1	312.4
36	27.14	5.6	311.3
40	27.18	5.8	313.4
44	27.14	5.6	314.2
48	27.31	6.3	313.9

Rig No. 2, with 5 metal specimens in test oil sump
Sump temp, °F 500
Oil in temp, °F 489
Bearing temp, °F 588*
Air flow to bearing machine, cfm 0.35
Air flow to test oil sump, cfm 0

*Test bearing held at this temperature average without application of external heat

TABLE XXV
GENERAL PROPERTIES OF PR-143 BASE GREASES

Evaluation	78-7% PR-143P 21.3% Ammelite	81.0% PR-143P 19.0% Ammelite	76.0% PR-143U 24.0% Ammelite	70.0% PR-143U 30.0% Teflon 120, PEP	90.0% PR-143, Lot I 10.0% Spherical BN	77.0% PR-143, Lot I 23.0% Ammelite	70.0% PR-143, Lot I 30.0% Teflon 120, PEP	93.0% PR-143, Lot I 7.0% Spherical BN
Uncoated penetration	260	383	319	290	329	309	288	303
Wetted penetration	275	391	348	315	346	324	326	339
Dropping point, °F	—	285	385	—	—	350	415	—
Evaporation, 22 hrs., % Wt loss @ 400°F	—	—	0.6	—	—	—	—	—
@ 450°F	—	—	1.4	—	—	—	—	—
@ 600°F	—	46	—	—	—	—	—	—
Separation, % @ 550°F	—	—	17.2	—	—	28.3	—	—
@ 600°F	—	34	24.3	—	—	—	—	—
LOX Compatibility, threshold value, ft-lb	—	—	+ 70	—	—	+ 70	+ 80	+ 70
Pope spindle, 204 bearing, 10,000 RPM, 5 lb @ 400°F	—	—	—	—	146	—	—	2432
@ 450°F	—	—	—	—	—	—	—	1265
@ 500°F	1740	—	1600 +	1635	—	700	—	328
@ 550°F	240	—	300	220	—	—	—	11
@ 600°F	140	120	128	—	—	—	—	3
Pope spindle, 204 bearing, 10,000 RPM, 25.50 lb @ 600°F	—	186	224	—	—	200	—	—
Pope spindle, 204 bearing, 20,000 RPM, 5 lb @ 600°F	—	60	—	—	—	—	—	—
Pope spindle, 204 bearing, 30,000 RPM, 5 lb @ 600°F	—	21	—	—	—	—	—	—
Pope spindle, R-4 bearing, 10,000 RPM, 3 lb, 10 ⁻⁷ , 400°F	—	—	1400	—	—	—	—	—
Pope spindle, R-4 bearing, 10,000 RPM, 5 lb, 10 ⁻⁷ , 400°F	—	—	595	—	—	—	—	—

TABLE XXVI

General Properties of PR-240 Grease

Base Oil	PR-143AC
Viscosity, cs. @ 100°F.	285
Viscosity, cs. @ 210°F.	25.5
Pour point, °F	-20
Thickener	"Vydax 1000 Fluorocarbon Telomer Dispersion
Thickener, weight %	14.2
Color	White
Texture	Buttery
ASTM Penetration, mm./10 @ 77°F.	
Unworked	266
Worked 60 strokes	275
NLGI Grade	2
Mechanical Stability	
ASTM Pen. after 100,000 strokes	311
ASTM Pen. after 6-hr. Shell Roll Test	315
Evaporation, FTMS ^a 791-351, Wt. % loss	
22 Hours @ 400°F.	1.6
22 Hours @ 500°F.	4.1
Water Resistance, FTMS 791-3252, % loss @ 100°F.	1.6
Load Carrying Cap., FTMS 791-6503	
Mean Hertz Load, kilograms	> 60
High Temperature Bearing Performance, FTMS 791-333	
Hours to failure @ 500°F.	> 2000
Hours to failure @ 550°F.	> 500
Dielectric Breakdown Voltage, ASTM D-877, kilovolts	43.6
Oxidation Stability, ASTM D-942, psig.oxygen pressure drop in 600 hrs. @ 210°F.	0
Copper Corrosion, FTMS 791-5309	Pass
Shock Sensitivity, USAF Spec. Bulletin 527	
Liquid oxygen impact test	Pass
Nitrogen tetroxide impact test	Pass

a. Federal Test Method Standard Number 791

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<p>A new class of high temperature fluids, designated by Du Pont code PR-143, has been extensively investigated to determine its potential in the area of advanced lubrication and energy transfer. The PR-143 fluids have exhibited excellent high temperature oxidative stability, a broad fluid range, good lubricity, and a high degree of fire-resistance; thereby, making them promising candidates for aerospace systems of the future. In the design of future systems some shortcomings of these perfluorinated polymeric materials must be considered; namely, corrosion of certain metal alloys at high temperatures and their relatively high density. (U)</p>		

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PR-143 Compatibility with Metals						
PR-143 Base Greases						
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